X-ray Characterization

Synchrotron radiation sources provide intense beams of X-rays for leading-edge research in a broad range of scientific disciplines. The Synchrotron Radiation Program at NIST/MSEL includes the development and operation of beam stations at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory, and at the Advanced Photon Source (APS) at Argonne National Laboratory. NIST is a partner at the APS with the University of Illinois at Urbana/Champaign, Oak Ridge National Laboratory, and UOP, in a collaboration called UNICAT (collaborative access team). The emphasis is on microstructure characterization, where NIST scientists and researchers from industry, universities and other government laboratories perform state-of-the-art measurements on advanced materials.

Scientific studies currently underway include microstructure characterization of ceramics and plasma-sprayed ceramic coatings, crystal perfection of basic and applied materials, microstructure evolution as a function of deformation, and the atomic-scale and molecular-scale structures at surfaces and interfaces.

Active programs at the NSLS include the operation of the X23A2 beam station where highly monochromatic X rays in the energy range 4.9 keV to over 30 keV are provided for X-ray absorption fine structure (XAFS) and diffraction anomalous fine structure (DAFS) measurements. These techniques enable the solution, for example, of the microscopic structures of technologically important strained thin films and rare earth doping of ceramic matrix composites. Another significant program at the NSLS is the operation of the soft-X-ray materials end station on beam line U7A, where X-ray photoemission spectroscopy and X-ray absorption spectroscopy are used to study the structure and chemistry of surfaces and bulk materials either in vacuum or under atmospheric reaction conditions. Recent studies include *in situ* catalyst characterization and an investigation of magnetic hard disk lubricant chemistry.

The first UNICAT APS beam line became operational November 1, 1999. It incorporates the newest technology, which enables NIST scientists to significantly improve our ultra-small-angle x-ray scattering, *in situ* X-ray topography, real-time x-ray microscopy, and X-ray absorption fine structure (XAFS) capabilities. It also offers opportunities for cutting-edge experiments in structural crystallography and time-resolved scattering, surface / interface scattering, diffuse scattering, and magnetic scattering. We anticipate extending our present portfolio of characterization capabilities to include an even wider range of materials measurements.

Experimental capabilities include:

- The brilliance at the APS makes it possible for the first time to monitor surfaces and interfaces *in situ* during MBE or CVD;
- Our sensitivity is increased by a factor of 100 in ultra-small-angle x-ray scattering from ceramics, coatings, biological materials and polymers;
- Resolution for imaging of defects in semiconductor crystals, window and dome materials, and superconducting crystals reach approximately 1 μm;
- Time resolved structural studies during melting or phase transitions in crystalline materials are now possible;
- Diffuse x-ray scattering can be used to determine structures and lattice imperfections in ceramics, metals, semiconductors, and superconductors;
- An extension of ultra-small-angle X-ray scattering (USAXS) into USAXS imaging is currently under development.

Future opportunities for development include the possibility of becoming a part of UNICAT-II: a synchrotron X-ray micro-beam capability for probing materials at the sub-micrometer level. High-throughput parallel detection X-ray diffraction and X-ray fluorescence on this instrument would offer a unique opportunity for combinatorial materials science. Additional advantages include the possibility of characterizing sub-micrometer strain and stress and selected interface structures. Commissioning exercises on a prototype instrument are currently underway to evaluate the final design.

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